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(54) ALUMINUM FLAKE PIGMENT, COATING COMPOSITION CONTAINING THE SAME AND COATING FILM

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain an aluminum flake pigment high in mechanical shear-resistance and excellent in brightness by making the pigment include an aluminum flake having a specific mean thickness, mean particle size, aspect ratio and uniformity number.

SOLUTION: This pigment is obtained by making it include an aluminum flake with the following properties: a mean thickness (t) (resulted from the steps of; adding 1 g of stearic acid to 1 g of aluminum flake; heating the mixture together with a little amount of xylene to thereby perform a leafing treatment; powdering it by acetone for measuring its water surface diffusion area; and calculating from the equation $t=4,000/\text{the water surface diffusion area}$) = 0.2 to 0.7 μm ; a mean particle size (D50) (by the laser diffraction method) = 4 to 20 μm ; an aspect ratio (D50/t)=15 to 50; and a uniformity number (n) (obtained by determining a particle size distribution on an area basis by using a laser diffraction type particle size distribution detector, plotting the cumulative distributions corresponding to the obtained respective particle sizes on a Rosin- Rammler diagram, translating the resultant line, and drawing an extrapolation line from the pole) ≥ 2.4 .

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CLAIMS

[Claim(s)]

[Claim 1] The aluminium flake pigment with which an aluminium flake is characterized by being average thickness (t)0.2-0.7micrometer, the mean particle diameter (D50) of 4-20 micrometers, aspect ratios (D50/t) 15-50, and (distribution constant n) ≥ 2.4 and brightness excelled [pigment] in circulation-proof nature highly.

[Claim 2] The aluminium flake pigment according to claim 1 whose brightness of an aluminium flake is 140 or more.

[Claim 3] The coating constituent which contains the aluminium flake pigment of 1 - 20 weight section according to claim 1 or 2 to the resin (solid content) 100 weight section.

[Claim 4] The coating constituent which contains an aluminium flake pigment according to claim 1 or 2, a fatty-acid AMAIDO wax, cellulose acetate butylate, and the transparence or the translucent inorganic bulking agent of 1 - 20 weight section to the resin (solid content) 100 weight section.

[Claim 5] The paint film which painted the coating constituent according to claim 3 or 4, and was formed.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the aluminium flake pigment for metallic paints especially brightness excelled [pigment] in the resistance (circulation [-proof] nature: Circulation Resistance) over the so-called circulation in a painting process highly about an automobile, a two-flower vehicle, the other aluminium flake pigments used for industrial paint, the coating constituent containing it, and a formation paint film.

[0002]

[Description of the Prior Art] The aluminium flake is used as a pigment for coatings which gives a premium grade metallic feeling conventionally. In case the coating containing an aluminium flake is generally actually painted, it is necessary to circulate a coating with stirring or a gear pump, or a plunger pump so that a pigment may not sediment. On the occasion of such stirring and circulation, by the intense turbulent flow a stirring aerofoil, near a pump, or inside piping, an excessive impact is added to an aluminium flake and the deformation and fracture follow. A result of the paint film by the coating which used deformation and the fractured aluminium flake pigment brings about the difficulty of the fall of whenever [luminosity], or lightness.

[0003] As advanced technology relevant to this technical problem, although patent No. 2090590 and 2506609 can be illustrated, these try solution of the technical problem concerned by limiting the shape factor of an aluminium flake to the specific range. However, in order to limit the shape factor of an aluminium flake to the specific range, the fall of the parallel array nature of the flake in reduction of the number of flakes in a paint film, polish extent with an inadequate flake front face, or a paint film with required consequently thickening flake thickness in each particle diameter etc. is concluded, it is still harder to escape the fall of admiration whenever [luminosity / of a paint film], and the result of a blacking wash could not fully be avoided.

[0004] Therefore, this invention aims at offer of the coating and film former in which whenever [luminosity] was moreover strongly excellent in mechanical Shache.

[0005]

[Means for Solving the Problem] Said technical problem may be effectively solved by the aluminium flake pigment containing average thickness (t) 0.2-0.7 micrometer, the mean particle diameter (D50) of 4-20 micrometers, aspect ratios (D50/t) 15-50, and the aluminium flake that is (distribution constant n) ≥ 2.4 . It will be as follows if the above-mentioned description of the aluminium flake of this invention etc. is explained in full detail.

[0006] 1. The very fine particle has adhered on the surface of a flake in many cases, especially the aluminium flake of mean particle diameter of the configuration former of a flake (example of comparison 2 reference shown in Figs. 3 and 4) is [this] remarkable in a flake 25 micrometers or less. Although it does not break away in the condition in the usual coating, under strong Shache, such as circulation, it breaks away, and this very fine particle causes black-discoloration-izing, when it is made into a paint film. Furthermore, adhesion of a very fine particle is one of the causes which check surface smooth nature, and brings about the fall of whenever [luminosity / of an aluminium flake]. Surface smooth nature and surrounding smoothness have the role important for the circulation-proof nature of a coating. That is, the flake which is not [that the circumference is notched and] smooth tends to be

transformed or fractured by mechanical Shache, and is concluded in a color change remarkable as a result.

[0007] If these are concretely explained with reference to an accompanying drawing (the example 3 shown in Figs. 1 and 2, example 2 of a comparison shown in Figs. 3 and 4 at a list), it will set in this invention article (example 3). The numbers of very fine particle adhesion of the example 2 of a comparison which the numbers of very fine particle adhesion are about 0.3 averages / flake, and is elegance conventionally to front-face and edge smooth nature being very good are about five or more averages / flake, and its front-face and edge smooth nature is remarkably poor. With the aluminium flake pigment of this invention, below two piece / flake are desirable.

[0008] 2. If mean particle diameter, an aspect ratio, and distribution constant mean particle diameter serve as a result of the iris of the eye by mixing of microscopic grains in less than 4 micrometers and 20 micrometers is exceeded, by the paint film thickness usually painted, the smooth nature of a paint film will worsen and image clarity will be spoiled by ejection (BUTSU) of a big and rough particle.

[0009] An aspect ratio is a factor which affects circulation-proof nature, if 50 is exceeded, flattening will progress and cutting and deformation of a flake will produce it by Shache at the time of circulation. Under such a phenomenon, the array of the flake in the inside of a paint film is confused remarkably, and photoluminescent [of a paint film] is spoiled. On the other hand, less than by 15, the parallel array of the flake in the inside of a paint film is not acquired, but photoluminescent [of a paint film] is spoiled similarly.

[0010] Less than by 2.4, particle size distribution become large and a distribution constant reduces lightness whenever [luminosity / of a paint film]. Moreover, the remarkable improvement in circulation-proof nature is obtained by equalizing particle size distribution under each grain size and thickness.

[0011] The distribution constant n of this invention searches for the particle size distribution of area criteria using a laser diffraction type particle-size-distribution measuring device, draws the line which will plot the cumulative distribution for every obtained particle diameter in a rosin-RAMURA diagram, will carry out the parallel displacement of the straight line, and will be removed from the pole (Pol P), and asks for n .

[0012] 3. The greatest description of brightness metallic coating finishing of a flake is a feeling of a metal which an aluminium flake has, therefore it is most important for it practically to raise own brightness of an aluminium flake.

[0013] Conventionally, in order to raise the brightness of an aluminium flake in elegance, to piece[of phosphorus]-ize more has been tried, but since such piece[of phosphorus]-izing is equivalent to enlarging an aspect ratio, it becomes the cause of reducing circulation-proof nature.

[0014] In this invention, it succeeded in raising the brightness of an aluminium flake by making the front face of an aluminium flake smooth, and smoothing a surrounding configuration.

[0015] It has the higher efficacy it not only supposes that it is possible the result which the flake moderately piece[of phosphorus]-ized in this way although this invention could offer 140 or more aluminium flakes for the first time excelled in brightness (L15 value) as metallic paint color, but that it can use it in straight silver (silver color by the aluminum simple substance), or light-coloring color (metallic color colored by organic pigment of light-coloring color) paint.

[0016] 4. In a coating constituent coating constituent, to the resin solid content 100 weight section, there will be very little abundance of a flake [in / in an aluminium flake pigment / a paint film], and it will lack photoluminescent [as metallic coating] under in 1 weight section. When 20 weight sections are exceeded, the flake content in a paint film becomes excessive, not only image clarity falls, but the smooth nature is spoiled by ejection of the flake on the front face of a paint film, and the possibility of the exfoliation in a layer arises according to it.

[0017] The resin for which the resin used for this invention is generally used as an object for coatings is used. For example, acrylic resin, polyester resin, alkyd resin, a fluororesin, etc. can be mentioned, it usually mixes with cross linking agents, such as amino resin and a block poly isocyanate compound, and use is presented. Moreover, these resin can also be used not only combining one sort but combining two sorts or more. In addition, the lacquer and 2 liquid type polyurethane resin which can usually be hardened by desiccation, silicone resin, etc. are used.

[0018] 5. In additive usual and metallic coating, atomized coating Myst reaches a coated object and

there is contraction of the paint film by fusion of a coating drop and volatilization of a solvent. The parallel array is attained in the phase of contraction according [the aluminium flake blended with the coating] to fusion of a coating, and volatilization of a solvent. That is, the coating nonvolatile matter on the coated object painted in the meantime goes up, viscosity also increases, and the array of an aluminium flake is determined. Volatile matter consists of solvent kinds (an aromatic series system, a ketone system, an ester system, glycol system, etc.) used, and affects the solubility of the resin of a coating, and workability.

[0019] In this invention, while delaying the viscous rise by volatilization of a solvent by addition of an additive, distribution of the aluminium flake in the inside of a paint film is made easy, and the parallel array property can be improved.

[0020] As an additive, more remarkable effectiveness is acquired by adding three sorts of a fatty-acid AMAIDO wax, cellulose acetate butylate and transparency, or a translucent inorganic bulking agent to coincidence.

[0021] Although usually used as a sedimentation inhibitor or a dripping inhibitor as a thixotropic agent in a metallic paint, if a fatty-acid AMAIDO wax is independent, the purpose which carries out the parallel array of the aluminium flake in a paint film has little effectiveness. A fatty-acid AMAIDO wax has desirable 5 - 15 weight section to resin solid content. If it separates from this range, the effectiveness of carrying out the parallel array of the aluminum flake pigment in a paint film will be hard to be acquired notably. Since cellulose acetate butylate has the effectiveness of viscous control, although effectiveness is in the stacking tendency of an aluminium flake, in the case of acrylic melamine system baking finish, the addition is restricted, and if independent, it cannot be said that sufficient effectiveness is acquired. 2 - 8 weight section of cellulose acetate butylate is desirable to resin solid content. If it separates from this range, the directing effect of an aluminum flake will be hard to be acquired notably. Although it was known that the brightness of a paint film will be improved by adding boron nitride and a barium sulfate also about an inorganic bulking agent, since all needed to make [many / very] the loadings, the use had a limit. Even if it is an inorganic bulking agent, using it in order to reduce the brightness of an aluminium flake should avoid the opaque thing, and it is [the thing] desirable. [of transparency or a translucent thing]

[0022] As transparency or a translucent inorganic bulking agent, an aluminum silicate, a calcium carbonate, precipitated barium sulphate, etc. can be illustrated.

[0023] Transparency or a translucent inorganic pigment has desirable 3 - 10 weight section to resin solid content. If it separates from this range, it will be hard to discover the brightness of an aluminum flake notably.

[0024] Other flake-like pigments, a color pigment, various kinds of additives, etc. can be shared if needed as other pigments used for the coating of this invention. As a flake-like pigment, tabular ferrous oxide, a phthalocyanine flake, graphite, a titanium-dioxide covering mica, a coloring mica, etc. can be mentioned, and these are used in the amount of extent which does not bar photoluminescent [of an aluminum flake pigment]. What is regularly used for coatings from the former is used, for example, a color pigment can mention an azo lake system pigment, phthalocyanine pigment, an indigo system pigment, a peri non system pigment, a perylene system pigment, a kino FUTARON system pigment, a dioxazine system pigment, the Quinacridone system pigment, an isoindolinone system pigment, a metal complex pigment, etc. as an organic system, and the chrome yellow, Synthetic Ochre, red ocher, carbon black, a titanium dioxide, etc. are mentioned as an inorganic system. The addition of a color pigment is set as arbitration according to the hue of paint color.

[0025] 6. as the methods of application, such as the method of application, it is based on air gun paint of common use, and electrostatic coating -- two quart of law etc. is suitable 1 BEKU. Furthermore, as paint color, an aluminium flake independent silver color, the metallic color colored by the organic pigment can be illustrated as a suitable thing.

[0026] The measuring method of the various characteristic values of the aluminium flake in measuring method this invention of various characteristic values is as follows.

[0027] 1. It is based on the laser diffraction method using laser DI contact sense non-isolated SALD1100 by mean-particle-diameter (D50) Shimadzu.

[0028] 2. 1g of stearin acid is added to average thickness (t) aluminium flake 1g, and it heats and leafing--ization-processes with a small amount of xylene, powder-ize with an acetone, measure a water

surface diffusion area, and compute thickness by the following formula (Aluminum Paint and Powder, J.D.Edwards and I.Wray, third edition, Reinhold Publishing Corp., New York (1955), and pages 16-22).

[0029]

average thickness (t:micrometer) = $4000 / \text{water surface diffusion area (cm}^2/\text{g)}$ 3. brightness (L15 value) Creation of a paint plate Aluminium flake pigment Net aluminum 1.5g Acryl lacquer * 48.5g (trade name [by * :Nippon Paint Co., Ltd.] auto -- clear) The above-mentioned coating is polished, and it applies to a glass plate with a 4-mil doctor blade at homogeneity, and dries at a room temperature.

[0030] L15 (L value of 30 degrees of incident angle 45-angle of reflection) is measured for the colorimetry approach aforementioned paint plate with X-Rite MA-68 mold (product made from X-Rite Incorporated).

[0031] (Note) Transparence acryl lacquer was used as a vehicle and it painted with the doctor blade as the method of application in which the parallel array of a flake is possible so that the brightness which the aluminium flake itself has could measure as much as possible.

[0032] 4. It is based on the observation using the configuration scanning electron microscope of a flake. [0033]

5. Circulation-proof nature Coating combination AKURIDIKKU A-322 240g You van by Dainippon Ink & Chemicals, Inc. 20SE-60 50g Aluminium flake pigment by Mitsui Chemicals, Inc. net aluminum The 24g above-mentioned coating (16 weight sections aluminium flake : to the resin solid content 100 weight section) is prepared, and thinner adjusts viscosity to Ford cup # 4 or 20 seconds. the coating before => circulation (coating A) -- 300g of this coating was moved in the jacketed vessel for circulating cooling waters, and it stirred for 1 hour with the agitator which has the stirring aerofoil of a turbine blade. Spray painting of the coating (coating B) above-mentioned coatings A and B after => circulation was carried out so that dry paint film thickness might be set to about 15 micrometers on a surface treatment steel plate with an automatic coating machine. Subsequently, 140 degrees C was able to be burned after paint for 30 minutes so that dry film thickness might be set to 35 micrometers by the wet-on-wet method on a paint film in a clear coat (an acrylic / melamine resin system coating "the super rack 0:150", Nippon Paint make). (wet-on-wet: Be burned after metallic coating approach which carries out a clear coat without desiccation, and it can be burned and is dried) L15 is measured for the colorimetry approach above-mentioned paint plate with said X-Rite MA-68 mold. Circulation-proof nature (CR nature) is computed by the degree type.

[0034] If it is 80% or more of CR nature (%) =(whenever [luminosity / of the paint film before circulation] whenever [luminosity / of the paint film after L15/circulation] L15) x100 evaluations, it is the range which can be used practically convenient.

[0035]

[Effect of the Invention] This invention offers the new aluminium flake pigment, coating constituent, and paint film in which whenever [luminosity] was moreover strongly excellent in mechanical Shache.

[0036]

[Example]

Example 1 Ball mill Bore of 500mm phi, die length of 180mm Raw material D50 4.5-micrometer spherical powder 800g Mineral spirit 5000ml Oleic acid 100g Rotational frequency 34rpm Operation time 5:30 (5 hours and 30 minutes)

Screen 400 meshes (two steps)

10% of very fine particle was cut by the decantation, and solid liquid separation was carried out with the filter, and the cake was mixed for 30 minutes and it considered as the aluminium flake pigment (65% of heating residues).

[0037] Example 2 raw material D50 7.0-micrometer spherical powder grinding time amount The example 3 raw material same except the 6:00 above as an example 1 D50 6.5-micrometer spherical powder grinding time amount The example 4 raw material same except the 4:30 above as an example 1 D50 5.5-micrometer spherical powder grinding time amount The example 5 raw material same except the 6:30 above as an example 1 D50 5.0-micrometer spherical powder grinding time amount It is the example 6 same raw material as an example 1 except the 6:00 above. D50 The example of comparison 1 bore of 500mm same except the 8.7-micrometer spherical powder grinding time amount 5:00 above as an example 1, In a ball mill with a die length of 180mm, 1.0kg (Toyol America, product X-81 made from Inc.) of atomized aluminium spherical powder, It was filled up with the compound which consists

of a mineral spirit 5.0l and 500g of oleic acid, and half[3 hours and]-operated by 38rpm (about 64% of critical speed) using with a diameter of 2mm steel ball 50kg.

[0038] The slurry after grinding termination and in a mill was probed by the mineral spirit, it applied to the oscillating screen of 150,350,400 meshes (37 micrometers) one by one, and solid liquid separation of the passed slurry was carried out with the pan filter. The obtained cake (NV85%) was moved in the kneader mixer, it kneaded for 1 hour, and the aluminum flake pigment (NV80%) was obtained.

[0039] Example of comparison 27640NS (Toyo Aluminium make)

Example of comparison 3TF3645 (silver-line company make)

Examples 1-6 and the examples 1-3 of a comparison evaluated circulation-proof nature by coating combination of a publication to the above-mentioned 5.

[0040] The aluminium flake pigment of example 11 example 5 was used, and the coating for a circulation-proof nature test was created.

[0041] However, the following additive was added further.

[0042]

CAB-381-05 10g Eastman Chemical Japan, Inc.

Flow non SP-1000 3g Kyoeisha Chemistry

Aluminium silicate ASP-200 5g It replaced with the aluminium flake pigment of en gel hard company example 12 example 5, and the coating for a circulation-proof nature test was created like the example 11 except having used the aluminium flake pigment of an example 6.

[0043] The aluminium flake pigment of example 13 example 6 was used, and the coating for a circulation-proof nature test was created.

[0044] However, the additive of an example 11 added only the following.

[0045]

CAB-381-05 10g Eastman Chemical Japan, Inc.

[0046]

[Table 1]

表1 アルミニウムフレークの特徴とCR性について

試料	D ₅₀ (μm)	平均厚み (μm) t	D ₅₀ /t	フレークの 輝度L _{1.5}	均等数 n	フレークの状態			CR性 (%)
						表面	端部	付着	
実施例1	9.0	0.21	42.8	140.1	2.5	◎	◎	◎	97
実施例2	14.6	0.30	48.7	149.2	2.4	◎	◎	◎	97
実施例3	16.6	0.62	26.8	152.3	2.4	◎	◎	◎	98
実施例4	11.7	0.25	46.8	140.4	2.9	◎	◎	◎	88
実施例5	12.5	0.32	39.1	149.5	2.7	◎	◎	◎	86
実施例6	17.4	0.51	34.1	155.2	2.7	◎	◎	◎	83
比較例1	34.9	2.70	12.9	134.9	2.0	◎	◎	○	95
比較例2	19.0	0.26	73.1	158.2	2.2	△	×	×	69
比較例3	17.2	0.80	21.5	132.5	2.0	○	○	△	90

表面：表面の平滑性

端部：端部のなめらかさ

付着：微粒子の付着

◎ ○ △ ×
良 ←————→ 悪

[0047]

[Table 2]

表2 塗料添加剤による塗膜の光輝性への影響

試料	D ₅₀ (μm)	平均厚み (μm) t	D ₅₀ /t	フレークの 輝度L _{1.5}	均等数 n	フレークの状態			CR性 (%)	塗膜の 光輝度L _{1.5} (*)
						表面	端部	付着		
実施例5 (比較用)	12.5	0.32	89.1	149.5	2.7	◎	◎	◎	86	128
実施例6 (比較用)	17.4	0.51	34.1	155.2	2.7	◎	◎	◎	83	136
実施例11	12.5	0.32	39.1	149.5	2.7	◎	◎	◎	86	139
実施例12	17.4	0.51	34.1	155.2	2.7	◎	◎	◎	83	153
実施例13 (比較用)	17.4	0.51	34.1	155.2	2.7	◎	◎	◎	83	141

(*)：サーキュレーション前の光輝度

[Translation done.]